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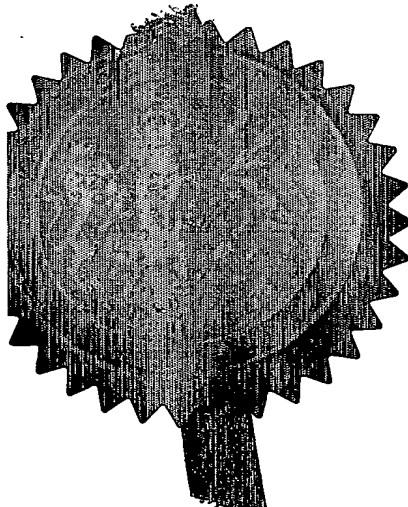
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Dated 9 April 2003

*Stephen Horrell*



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## Request for grant of a patent

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20 APR 2002

NEWPORT

The Patent Office

Cardiff Road  
Newport  
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Your reference

10992P6 GB/AB

Patent application number  
*(The Patent Office will fill in this part)*

0209093.4

20 APR 2002

Full name, address and postcode of the or of each applicant (*underline all surnames*)Reckitt Benckiser N.V.  
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07921075005

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Patents ADP number (*if you know it*)

If the applicant is a corporate body, give the country/state of its incorporation

Title of the invention

Improvements in or Relating to Compositions

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UNITED KINGDOM"Address for service" in the United Kingdom to which all correspondence should be sent (*including the postcode*)Patents ADP number (*if you know it*)

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Country

Priority application number  
(*if you know it*)Date of filing  
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If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application  
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Yes

- a) any applicant named in part 3 is not an inventor, or
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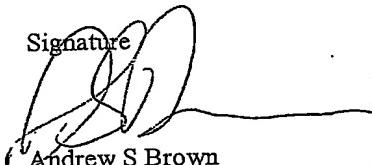
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11.

I/We request the grant of a patent on the basis of this application.

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Andrew S Brown

19 April 2002

12. Name and daytime telephone number of Person to contact in the United Kingdom

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# DUPLICATE

10992P6GB

1

## IMPROVEMENTS IN OR RELATING TO COMPOSITIONS

The present invention relates to a water-soluble container  
5 comprising at least one compartment prepared from a  
thermoformed sheet of hydroxy propyl methyl cellulose (HPMC)  
and to a process for preparing such a container.

It is known to package chemical compositions, particularly  
10 those which may be of a hazardous or irritant nature, in  
films, particularly water soluble films. Such containers  
can simply be added to water in order to dissolve or  
disperse the contents of the container into the water.

15 For example, WO 89/12587 discloses a package which comprises  
an envelope of a water soluble material which comprises a  
flexible wall and a water-soluble heat seal. The package  
may contain an organic liquid comprising, for example, a  
pesticide, fungicide, insecticide or herbicide.

20 WO 92/17382 discloses a package containing an agrochemical  
comprising a first sheet of non-planar water-soluble or  
water-dispersible material and a second sheet of water-  
soluble or water-dispersible material superposed on the  
25 first sheet and sealed to it.

EP-A-1,126,070 relates to laundry additive sachets which may  
be made from HPMC film. No detailed discussion is given on  
how the sachets may be prepared. The single Example provides  
30 no details. Paragraph 0013 simply states that they can be  
prepared by known methods in the art, specifically by first

cutting an appropriately sized piece of film or sheet, folding the sheet to form the necessary number and size of compartments and sealing the edges, for example by heat sealing.. There is no reference to thermoforming of HPMC.

5

A well known problem with sachet made from poly(vinyl alcohol) (PVOH) is that it may be easily cross-linked and, subsequently, made less soluble by certain compounds. A known cross-linking agent are perborate bleaching compounds 10 commonly found in fabric detergents. Cross-linking may be avoided by eliminating unsuitable compounds from the sachet. When a sachet is used in addition to a main detergent added 15 to the wash, as a wash booster/wash additive, then cross-linking agents may be commonly found in any number of main detergents found on the market. There is a need to use films that do not cross link, especially for wash additive products.

A suitable film for making sachets is HPMC. However, HPMC is 20 known to have poor physical properties, such as a very low tear strength, which previously has meant that this film is thought to be unsuitable for use in standard thermoforming processes, where stretching is an inevitable outcome of the process.

25

Surprisingly, we have succeeded in thermoforming HPMC by careful selection of the conditions, both with carrier technology (such as by the use of a PET film, ideally 200-250 micron thick, which is passed through the thermoforming 30 machine underneath the HPMC film (see GB2362868 for a more detailed explanation of the technique), and without the use

of carrier technology. The reason for using the carrier technology is to help the film to maintain its shape and to increase the physical strength of the sachet.

- 5 We succeeded in thermoforming HPMC of different thicknesses (from 75 to 120 micron), in all cases obtaining a good shape, with and without carrier technology, and without any tearing. We succeeded also in making an on-line lamination of HPMC (for example, with two films of HPMC 75 micron each  
10 thermoformed together). This is another method to increase the strength of the final capsule, see GB0201542.8 for a more detailed explanation of the on-line lamination technique.
- 15 Another important finding is that HPMC can be sealed easily and produces a seal with good resistance to breaking.

The present invention provides a water-soluble container comprising at least one compartment prepared from a  
20 thermoformed film of hydroxy propyl methyl cellulose (HPMC).

A further feature of the invention is a process for preparing a water-soluble container comprising at least one compartment which comprises:

- 25 a. feeding at least one HPMC film into a thermoforming machine;  
b. heating the at least one HPMC film to a temperature of 120 to 140°C, ideally 125 to 135°C, preferably for 1 to 10 seconds, ideally 2 to 6 seconds;

- c. simultaneously or subsequently forming the at least one heated HPMC film into a mould, which is preferably cooled (below room temperature, ideally below 20°C);
- d. filling the formed film pocket with at least one liquid or solid composition; and
- e. sealing the formed and filled film pocket with a water-soluble film, preferably a HPMC film.

In the process of the present invention at two or more compartments can be made provided. Each compartment may be a single compartment or comprise two or more individual compartments. For example each compartment may be separated by one or more dividing walls into two or more individual compartments.

The film may be a single film, or a laminated film as disclosed in GB-A-2,244,258. While a single film may have pinholes, the two or more layers in a laminate are unlikely to have pinholes which coincide. The layers in a laminate may be the same or different.

The film may be produced by any suitable process, for example by extrusion and blowing or by casting. At present generally only cast film is commercially available, such as from agents like Aquafilm (UK), Hartlebury, Worcestershire, DY10 4JB and produced by ENAK Redkiln Way Horsham Sussex England RH13 5QH. Other manufacturer include Cast of USA.

The thickness of the film used to produce the sachets is preferably 40 to 300 µm, more preferably 70 to 200 µm,

especially 80 to 160  $\mu\text{m}$ , more especially 90 to 150  $\mu\text{m}$  and most especially 90 to 120  $\mu\text{m}$ .

In a thermoforming process a film may be drawn down or blown  
5 down into a mould after it has been heated. Thus, for example, the film is heated to the thermoforming temperature using a thermoforming heater plate assembly, and then drawn down under vacuum or blown down under pressure into the mould. Plug-assisted thermoforming and pre-stretching the  
10 film, for example by blowing the film away from the mould before thermoforming, may, if desired, be used. The exact amount of vacuum or pressure and the exact thermoforming temperature used depend on the thickness and porosity of the film used.

15

A suitable forming pressure is, for example, 69 to 138kPa, especially 83 to 117 kPa. A suitable forming vacuum is 0 to 4 kPa, especially 0 to 2 kPa. A suitable time for the film to be held in the mould ("dwell time") is, for example, 0.4  
20 to 2.5 seconds, especially 2 to 2.5 seconds.

After the compartments have been formed, they are filled with the desired composition(s) which are intended to be released in an aqueous environment.

25

The compositions may be a fabric care, surface care or dishwashing composition. Thus, for example, they may be a dishwashing, water-softening, laundry or detergent composition, or a rinse aid. Such compositions may be  
30 suitable for use in a domestic washing machine. The compositions may also independently be a disinfectant,

antibacterial or antiseptic composition, or a refill composition for a trigger-type spray. Such compositions are generally packaged in total amounts of from 5 to 100 g, especially from 15 to 40 g. For example, a dishwashing 5 composition may weigh from 15 to 30 g, a water-softening composition may weigh from 15 to 40 g.

The compartment(s) may be completely filled or only partially filled. Each composition independently may be a 10 solid. For example, it may be a particulate or granulated solid, or a tablet. Each composition may also independently be a liquid, which may be thickened or gelled if desired. The liquid composition may be non-aqueous or aqueous, for example comprising less than or more than 5% or less than or 15 more than 10wt% total or free water. Desirably the compositions contain less than 80 wt% water.

Each composition may have more than one phase. For example each composition may comprise an aqueous composition and a 20 liquid composition which is immiscible with the aqueous composition. Each composition may also comprise a liquid composition and a separate solid composition, for example in the form of a ball, pill or speckles.

25 The compositions may be appropriately chosen depending on the desired use of the article. For example in a sachet comprising two or more compartments may contain the following:

30 1. laundry washing, the primary composition may comprise, for example, a detergent, and the secondary composition may

comprise a bleach, stain remover, water-softener, enzyme or fabric conditioner. The article may be adapted to release the compositions at different times during the laundry wash. For example, a bleach or fabric conditioner is generally released at the end of a wash, and a water-softener is generally released at the start of a wash. An enzyme may be released at the start or the end of a wash.

2. fabric conditioner, the primary composition may comprise a fabric conditioner and the secondary component may comprise an enzyme which is released before or after the fabric conditioner in a rinse cycle.

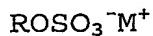
3. dish washing the primary composition may comprise a detergent and the secondary composition may comprise a water-softener, salt, enzyme, rinse aid, bleach or bleach activator. The article may be adapted to release the compositions at different times during the laundry wash. For example, a rinse aid, bleach or bleach activator is generally released at the end of a wash, and a water-softener, salt or enzyme is generally released at the start of a wash.

4. laundry additive. The primary composition may contain a bleaching system, preferably as a powder (e.g. sodium percarbonate, perborate or K or NaDIC potassium or sodium di-chloroisocyanurate, or KHSO<sub>3</sub> potassium persulfate or an activator, such as, phthalimidoperoxyhexanoic acid (PAP as supplied by Ausimont) or a nitrile quat., such as methylmorpholin aceto nitrile di-sulfate (example SOKALAN BM

G supplied by BASF). The secondary composition may contain a solid, liquid or gel based on enzyme and surfactants.

The ingredients of each composition depend on the use of the  
5 composition. Thus, for example, the composition may contain surface active agents such as an anionic, nonionic, cationic, amphoteric or zwitterionic surface active agents or mixtures thereof.

- 10 Examples of anionic surfactants are straight-chained or branched alkyl sulfates and alkyl polyalkoxylated sulfates, also known as alkyl ether sulfates. Such surfactants may be produced by the sulfation of higher C<sub>8</sub>-C<sub>20</sub> fatty alcohols.
- 15 Examples of primary alkyl sulfate surfactants are those of formula:



wherein R is a linear C<sub>8</sub>-C<sub>20</sub> hydrocarbyl group and M is a water-solubilising cation. Preferably R is C<sub>10</sub>-C<sub>16</sub> alkyl, for  
20 example C<sub>12</sub>-C<sub>14</sub>, and M is alkali metal such as lithium, sodium or potassium.

Examples of secondary alkyl sulfate surfactants are those which have the sulfate moiety on a "backbone" of the  
25 molecule, for example those of formula:



wherein m and n are independently 2 or more, the sum of m+n typically being 6 to 20, for example 9 to 15, and M is a water-solubilising cation such as lithium, sodium or  
30 potassium.

Especially preferred secondary alkyl sulfates are the (2,3) alkyl sulfate surfactants of formulae:



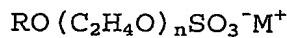
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for the 2-sulfate and 3-sulfate, respectively. In these formulae x is at least 4, for example 6 to 20, preferably 10 to 16. M is cation, such as an alkali metal, for example lithium, sodium or potassium.

Examples of alkoxylated alkyl sulfates are ethoxylated alkyl sulfates of the formula:

15



wherein R is a C<sub>8</sub>-C<sub>20</sub> alkyl group, preferably C<sub>10</sub>-C<sub>18</sub> such as a C<sub>12</sub>-C<sub>16</sub>, n is at least 1, for example from 1 to 20, preferably 1 to 15, especially 1 to 6, and M is a salt-forming cation such as lithium, sodium, potassium, ammonium, alkylammonium or alkanolammonium. These compounds can provide especially desirable fabric cleaning performance benefits when used in combination with alkyl sulfates.

25

The alkyl sulfates and alkyl ether sulfates will generally be used in the form of mixtures comprising varying alkyl chain lengths and, if present, varying degrees of alkoxylation.

30

Other anionic surfactants which may be employed are salts of fatty acids, for example C<sub>8</sub>-C<sub>18</sub> fatty acids, especially the sodium or potassium salts, and alkyl, for example C<sub>8</sub>-C<sub>18</sub>, benzene sulfonates.

5

Examples of nonionic surfactants are fatty acid alkoxylates, such as fatty acid ethoxylates, especially those of formula:



10

wherein R is a straight or branched C<sub>8</sub>-C<sub>16</sub> alkyl group, preferably a C<sub>9</sub>-C<sub>15</sub>, for example C<sub>10</sub>-C<sub>14</sub>, alkyl group and n is at least 1, for example from 1 to 16, preferably 2 to 12, more preferably 3 to 10.

15

The alkoxylated fatty alcohol nonionic surfactant will frequently have a hydrophilic-lipophilic balance (HLB) which ranges from 3 to 17, more preferably from 6 to 15, most preferably from 10 to 15.

20

Examples of fatty alcohol ethoxylates are those made from alcohols of 12 to 15 carbon atoms and which contain about 7 moles of ethylene oxide. Such materials are commercially marketed under the trademarks Neodol 25-7 and Neodol 23-6.5 by Shell Chemical Company. Other useful Neodols include Neodol 1-5, an ethoxylated fatty alcohol averaging 11 carbon atoms in its alkyl chain with about 5 moles of ethylene oxide; Neodol 23-9, an ethoxylated primary C<sub>12</sub>-C<sub>13</sub> alcohol having about 9 moles of ethylene oxide; and Neodol 91-10, an ethoxylated C<sub>9</sub>-C<sub>11</sub> primary alcohol having about 10 moles of ethylene oxide.

Alcohol ethoxylates of this type have also been marketed by Shell Chemical Company under the Dobanol trademark. Dobanol 91-5 is an ethoxylated C<sub>9</sub>-C<sub>11</sub> fatty alcohol with an average 5 of 5 moles ethylene oxide and Dobanol 25-7 is an ethoxylated C<sub>12</sub>-C<sub>15</sub> fatty alcohol with an average of 7 moles of ethylene oxide per mole of fatty alcohol.

Other examples of suitable ethoxylated alcohol nonionic 10 surfactants include Tergitol 15-S-7 and Tergitol 15-S-9, both of which are linear secondary alcohol ethoxylates available from Union Carbide Corporation. Tergitol 15-S-7 is a mixed ethoxylated product of a C<sub>11</sub>-C<sub>15</sub> linear secondary alkanol with 7 moles of ethylene oxide and Tergitol 15-S-9 15 is the same but with 9 moles of ethylene oxide.

Other suitable alcohol ethoxylated nonionic surfactants are Neodol 45-11, which is a similar ethylene oxide condensation products of a fatty alcohol having 14-15 carbon atoms and 20 the number of ethylene oxide groups per mole being about 11. Such products are also available from Shell Chemical Company.

Further nonionic surfactants are, for example, C<sub>10</sub>-C<sub>18</sub> alkyl 25 polyglycosides, such as C<sub>12</sub>-C<sub>16</sub> alkyl polyglycosides, especially the polyglucosides. These are especially useful when high foaming compositions are desired. Further surfactants are polyhydroxy fatty acid amides, such as C<sub>10</sub>-C<sub>18</sub> N-(3-methoxypropyl) glyciamides and ethylene oxide-propylene 30 oxide block polymers of the Pluronic type.

Examples of cationic surfactants are those of the quaternary ammonium type.

The total content of surfactants in the composition is  
5 desirably 60 to 95 wt%, especially 75 to 90 wt%. Desirably  
an anionic surfactant is present in an amount of 50 to 75  
wt%, the nonionic surfactant is present in an amount of 5 to  
50 wt%, and/or the cationic surfactant is present in an  
amount of from 0 to 20 wt%. The amounts are based on the  
10 total solids content of the composition, i.e. excluding any  
solvent which may be present.

The compositions, particularly when used as laundry washing  
or dishwashing compositions, may also independently comprise  
15 enzymes, such as protease, lipase, amylase, cellulase and  
peroxidase enzymes. Such enzymes are commercially available  
and sold, for example, under the registered trade marks  
Esperase, Alcalase and Savinase by Nova Industries A/S and  
Maxatase by International Biosynthetics, Inc. Desirably  
20 the enzymes are independently present in the primary or  
secondary compositions in an amount of from 0.5 to 3 wt%,  
especially 1 to 2 wt%, when added as commercial preparations  
they are not pure and this represents an equivalent amount  
of 0.005 to 0.5 wt% of pure enzyme.

25 The compositions may, if desired, independently comprise a  
thickening agent or gelling agent. Suitable thickeners are  
polyacrylate polymers such as those sold under the trade  
mark CARBOPOL, or the trade mark ACUSOL by Rohm and Haas  
30 Company. Other suitable thickeners are xanthan gums. The

thickener, if present, is generally present in an amount of from 0.2 to 4 wt%, especially 0.5 to 2 wt%.

Compositions used in dishwashing and laundry independently  
5 usually comprise a detergency builder. The builders counteract the effects of calcium, or other ion, water hardness encountered. Examples of such materials are citrate, succinate, malonate, carboxymethyl succinate, carboxylate, polycarboxylate and polyacetyl carboxylate  
10 salts, for example with alkali metal or alkaline earth metal cations, or the corresponding free acids. Specific examples are sodium, potassium and lithium salts of oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, C<sub>10</sub>-C<sub>22</sub> fatty acids and citric acid. Other examples are organic  
15 phosphonate type sequestering agents such as those sold by Monsanto under the trade mark Dequest and alkylhydroxy phosphonates. Citrate salts and C<sub>12</sub>-C<sub>18</sub> fatty acid soaps are preferred. Further builders are; phosphates such as sodium, potassium or ammonium salts of mono-, di- or tri-poly or  
20 oligo-phosphates; zeolites; silicates, amorphous or structured, such as sodium, potassium or ammonium salts.

Other suitable builders are polymers and copolymers known to have builder properties. For example, such materials  
25 include appropriate polyacrylic acid, polymaleic acid, and polyacrylic/polymaleic and copolymers and their salts, such as those sold by BASF under the trade mark Sokalan.

The builder is desirably present in an amount of up to 90  
30 wt%, preferably 15 to 90 wt%, more preferable 15 to 75 wt%, relative to the total weight of the composition. Further

details of suitable components are given in, for example, EP-A-694,059, EP-A-518,720 and WO 99/06522.

The compositions can also independently optionally comprise  
5 one or more additional ingredients. These include conventional detergent composition components such as further surfactants, bleaches, bleach enhancing agents, builders, suds boosters or suds suppressors, anti-tarnish and anti-corrosion agents, organic solvents, co-solvents,  
10 phase stabilisers, emulsifying agents, preservatives, soil suspending agents, soil release agents, germicides, pH adjusting agents or buffers, non-builder alkalinity sources, chelating agents, clays such as smectite clays, enzyme stabilizers, anti-limescale agents, colourants, dyes, 15 hydrotropes, dye transfer inhibiting agents, brighteners, and perfumes. If used, such optional ingredients will generally constitute preferably no more than 15 wt%, for example from 1 to 6 wt%, the total weight of the compositions.

20 Compositions which comprise an enzyme may optionally contain materials which maintain the stability of the enzyme. Such enzyme stabilizers include, for example, polyols such as propylene glycol, boric acid and borax. Combinations of 25 these enzyme stabilizers may also be employed. If utilized, the enzyme stabilizers generally constitute from 0.1 to 5 wt%, ideally 0.1 to 1 wt% of the compositions.

The compositions may independently optionally comprise  
30 materials which serve as phase stabilizers and/or co-solvents. Example are C<sub>1</sub>-C<sub>3</sub> alcohols such as methanol,

ethanol and propanol. C<sub>1</sub>-C<sub>3</sub> alkanolamines such as mono-, di- and triethanolamines can also be used, by themselves or in combination with the alcohols. The phase stabilizers and/or co-solvents can, for example, constitute 0 to 1 wt%,  
5 preferably 0.1 to 0.5 wt%, of the composition.

The compositions may independently optionally comprise components which adjust or maintain the pH of the compositions at optimum levels. The pH may be from, for  
10 example, 1 to 13, such as 8 to 11 depending on the nature of the composition. For example a dishwashing composition desirably has a pH of 8 to 11, a laundry composition desirable has a pH of 7 to 9, and a water-softening composition desirably has a pH of 7 to 9. Examples of pH  
15 adjusting agents are NaOH and citric acid.

The above examples may be used for dish or fabric washing. In particular dish washing formulations are preferred which are adapted to be used in automatic dish washing machines.  
20 Due to their specific requirements specialised formulation are required and these are illustrated below

Amounts of the ingredients can vary within wide ranges, however preferred automatic dishwashing detergent  
25 compositions herein (which typically have a 1% aqueous solution pH of above 8, more preferably from 9.5 to 12, most preferably from 9.5 to 10.5) are those wherein there is present: from 5% to 90%, preferably from 5% to 75%, of builder; from 0.1% to 40%, preferably from 0.5% to 30%, of  
30 bleaching agent; from 0.1% to 15%, preferably from 0.2% to 10%, of the surfactant system; from 0.0001% to 1%,

preferably from 0.001% to 0.05%, of a metal-containing bleach catalyst; and from 0.1% to 40%, preferably from 0.1% to 20% of a water-soluble silicate. Such fully-formulated embodiments typically further comprise from 0.1% to 15% of a 5 polymeric dispersant, from 0.01% to 10% of a chelant, and from 0.00001% to 10% of a deterotive enzyme, though further additional or adjunct ingredients may be present. Detergent compositions herein in granular form typically limit water content, for example to less than 7% free water, for better 10 storage stability.

Non-ionic surfactants useful in ADW (Automatic Dish Washing) compositions of the present invention desirably include surfactant(s) at levels of from 2% to 60% of the 15 composition. In general, bleach-stable surfactants are preferred. Non-ionic surfactants generally are well known, being described in more detail in Kirk Othmer's Encyclopedia of Chemical Technology, 3rd Ed., Vol. 22, pp. 360-379, "Surfactants and Detersive Systems", incorporated by 20 reference herein.

Preferably the ADW composition comprises at least one non-ionic surfactant. One class of non-ionics are ethoxylated non-ionic surfactants prepared by the reaction of a 25 monohydroxy alkanol or alkylphenol with 6 to 20 carbon atoms with preferably at least 12 moles particularly preferred at least 16 moles, and still more preferred at least 20 moles of ethylene oxide per mole of alcohol or alkylphenol.

30 Particularly preferred non-ionic surfactants are the non-ionic from a linear chain fatty alcohol with 16-20 carbon

atoms and at least 12 moles particularly preferred at least 16 and still more preferred at least 20 moles of ethylene oxide per mole of alcohol.

5 According to one preferred embodiment the non-ionic surfactant additionally comprise propylene oxide units in the molecule. Preferably this PO units constitute up to 25% by weight, preferably up to 20% by weight and still more preferably up to 15% by weight of the overall molecular  
10 weight of the non-ionic surfactant. Particularly preferred surfactants are ethoxylated mono-hydroxy alkanols or alkylphenols, which additionally comprises polyoxyethylene-polyoxypropylene block copolymer units. The alcohol or alkylphenol portion of such surfactants constitutes more  
15 than 30%, preferably more than 50%, more preferably more than 70% by weight of the overall molecular weight of the non-ionic surfactant.

Another class of non-ionic surfactants includes reverse  
20 block copolymers of polyoxyethylene and polyoxypropylene and block copolymers of polyoxyethylene and polyoxypropylene initiated with trimethylolpropane.

Another preferred non-ionic surfactant can be described by  
25 the formula:



wherein  $R^1$  represents a linear or branched chain aliphatic  
30 hydrocarbon group with 4-18 carbon atoms or mixtures thereof,  $R^2$  represents a linear or branched chain aliphatic

hydrocarbon rest with 2-26 carbon atoms or mixtures thereof,  $x$  is a value between 0.5 and 1.5 and  $y$  is a value of at least 15.

- 5 Another group of preferred nonionic surfactants are the end-capped polyoxyalkylated non-ionics of formula:



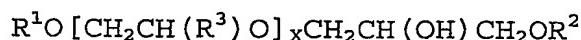
10 wherein  $R^1$  and  $R^2$  represent linear or branched chain, saturated or unsaturated, aliphatic or aromatic hydrocarbon groups with 1-30 carbon atoms,  $R^3$  represents a hydrogen atom or a methyl, ethyl, n-propyl, iso-propyl, n-butyl, 2-butyl or 2-methyl-2-butyl group,  $x$  is a value between 1 and 15 and,  $k$  and  $j$  are values between 1 and 12, preferably between 1 and 5. When the value of  $x$  is  $\geq 2$  each  $R^3$  in the formula above can be different.  $R^1$  and  $R^2$  are preferably linear or branched chain, saturated or unsaturated, aliphatic or aromatic hydrocarbon groups with 6-22 carbon atoms, where group with 8 to 18 carbon atoms are particularly preferred. For the group  $R^3$  H, methyl or ethyl are particularly preferred. Particularly preferred values for  $x$  are comprised between 1 and 20, preferably between 6 and 15.

25

As described above, in case  $x > 2$ , each  $R^3$  in the formula can be different. For instance, when  $x=3$ , the group  $R^3$  could be chosen to build ethylene oxide ( $R^3=H$ ) or propylene oxide ( $R^3=methyl$ ) units which can be used in every single order for 30 instance (PO)(EO)(EO), (EO)(PO)(EO), (EO)(EO)(PO), (EO)(EO)(EO), (PO)(EO)(PO), (PO)(PO)(EO) and (PO)(PO)(PO).

The value 3 for x is only an example and bigger values can be chosen whereby a higher number of variations of (EO) or (PO) units would arise.

- 5 Particularly preferred end-capped polyoxyalkylated alcohols of the above formula are those where k=1 and j=1 originating molecules of simplified formula:



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The use of mixtures of different non-ionic surfactants is particularly preferred in ADW formulations for example mixtures of alkoxylated alcohols and hydroxy group containing alkoxylated alcohols.

15

After the compartments have been filled, the compartments are closed by a lid. The lid may be of any form, so long as it is water-soluble.

- 20 The thickness of the film used for the lid may be less than the thickness of the film making up the compartment of the container because the film is not subjected to localised stretching in a thermoforming step, if thermoforming is used to form the compartments. It is also desirable to have a  
25 thickness which is less than that of the film used to form the first compartment to ensure a sufficient heat transfer through the film to soften the base web if heat sealing is used.

The thickness of the covering film is generally from 20 to 160  $\mu\text{m}$ , preferably from 40 to 100  $\mu\text{m}$ , such as 40 to 80  $\mu\text{m}$  or 50 to 60  $\mu\text{m}$ .

- 5 This film may be a single-layered film but is desirably laminated to reduce the possibility of pinholes allowing leakage through the film. The film may be the same or different as the film forming the first compartment. If two or more films are used to form the film comprising the  
10 second compartment, the films may be the same or different. Examples of suitable films are those given for the film forming the first compartment.

The lids are sealed to the compartments in order to enclose  
15 the compositions. Any method of sealing may be used. For example, the compartments and lids may simply be sealed by the application of pressure to the compartment or lid. It may be sealed to the compartment by any suitable means, for example by means of an adhesive or by heat sealing. Other  
20 methods of sealing include infra-red, radio frequency, ultrasonic, laser, solvent, vibration and spin welding. The seal desirably is water-soluble.

If heat sealing is used, a suitable sealing temperature is,  
25 for example, 185 to 205°C, for example 190 to 200°C. A suitable sealing pressure is, for example, from 250 to 600 kPa. Examples of sealing pressures are 276 to 552 kPa, especially 345 to 483 kPa or 400 to 800 kPa, especially 500 to 700 kPa depending on the heat sealing machine used.  
30 Suitable sealing dwell times are 0.4 to 2.5 seconds.

One skilled in the art can use an appropriate temperature, pressure and dwell time to achieve a seal of the desired integrity. While desirably conditions are chosen within the above ranges, it is possible to use one or more of these 5 parameters outside the above ranges, although it would might be necessary to compensate by changing the values of the other two parameters.

The containers may be produced in pairs, each unit of the 10 pair being joined by the folding portion. The containers may also be produced in strips of two, wherein the folding portion is the middle part of the strip between the lines of containers. The strips of containers may be used in the folding step as is, or individual pairs of containers, or 15 shorter strips, may be prepared by cutting the strips at appropriate points.

Desirably, however, the containers are produced in a two-dimensional array. It is possible, for example, to have an 20 array of up to 12 containers along one side and up to 10 containers along the second side. A suitable array size is four or six containers along one side, and four to eight containers along the other side. An especially preferred array size is eight containers along one side and six 25 containers along the other side. If desired the array can be cut to provide a smaller array of containers, a strip of pairs of containers, or individual pairs. Preferably, however, the array is used as is in the folding step.

30 Once the containers have been produced, they may be separated from each other by cutting the areas between them.

Alternatively, they may be left conjoined and, for example, perforations provided between the individual containers so that they can be easily separated at a later stage, for example by a consumer. If the containers are separated, the flanges 5 may be left in place. However, desirably the flanges are partially removed in order to provide an even more attractive appearance. Generally the flanges remaining should be as small as possible for aesthetic purposes while bearing in mind that some flange is required to ensure the 10 two films remain adhered to each other. A flange having a width of 1 mm to 8 mm is desirable, preferably 2 mm to 7 mm, most preferably about 5 mm.

The folding portion, which by this time has been folded, may 15 be retained in the containers. Desirably, however, it is at least partially removed, for example by trimming with a blade, to provide the containers with a more attractive appearance.

20 The containers of the present invention may have any desired shape.

The compartments may have the same or different size and/or shape. In general, if it is desired to have compartments 25 containing different quantities of components, the compartments have volume ratios of from 2:1 to 20:1, especially from 4:1 to 10:1. The pairs of compartments may have the same lid size and shape for adhering to each other. Alternatively they may have a different size and/or a 30 different shape. It is preferred that if the compartments have a different size, they have the same shape. In this

case the lid of the smaller compartment is adhered to only part of the lid of the larger compartment. Two or more smaller compartments can, if desired, be adhered to the lid of the larger compartment.

5

The containers produced by the process of the present invention, especially when used for a fabric care, surface care or dishwashing composition, may have a maximum dimension of 5 cm, excluding any flanges. For example, a

10 container may have a length of 1 to 5 cm, especially 3.5 to 4.5 cm, a width of 1.5 to 3.5 cm, especially 2 to 3 cm, and a height of 1 to 2 cm, especially 1.25 to 1.75 cm..

15 The containers may themselves be packaged in outer containers if desired, for example non-water soluble containers which are removed before the water-soluble containers are used.

#### THERMOFORMING HPMC

20

BOTTO M FILM 1 (M)	BOTTO M FILM 75	TOP FILM 75	CARRIER	HEAT TIME (s)	TEMP (°C)	TIME (s)	SEALIN G TEMP (°C) 195	SEALI NG TIME (s) 1.5
HPMC	HPMC	HPMC		4	130	1.5		
75	75	75						
HPMC 120		HPMC 75		4	130	1.5	195	1.5

HPMC 120		HPMC 75	PET 200 micron	4	130	3.5	188	1
HPMC 120		HPMC 75	PET 200 micron	4	130	3.5	195	
HPMC 75		HPMC 75	PET 250 micron	4	130	1.5	195	1.5
HPMC 75		HPMC 75		4	130	1.5	195	1.5
HPMC 100		HPMC 75		4	130	1.5	195	1.5

## SOLUBILITY OF HPMC CAPS

The solubility of HPMC capsules has been tested in Lab and

5 in real use conditions (washing machine).

Lab Test: method: 2 litres of Tap Water 20°C; stirring system at 220 rpm's.

Usually the test is done with water, with water+8% powder detergent, with water +8% boron containing powder detergent. Time of liquid release and time of complete dissolution of the film are registered.

Results: Time of liquid release is in line with PVOH (40-50 sec) and is not influenced by the type of detergent used (with or without boron).

Complete dissolution time is about 6-7 (depending on the thickness of the film) minutes and still is not influenced by the presence of boron in the detergent

(PVOH capsules in presence of boron containing detergent are not completely dissolved after 20 minutes).

Washing Machine Solubility test: the washing machine tested  
5 can be divided into 2 groups.

1. Conventional Washing machine: water loaded  $\cong$  18 lt.
  2. Fuzzy Logic Machine: water loaded  $\cong$  14 lt.

The tests were done combining the following variables:

- Temperature of the cycle: 30° C; 60 °C
  - Detergent: containing boron ; non containing boron
  - Detergent dose: 121,5 gr. (normal suggested dose);  
180 gr. (dose suggested for very dirty loaded); 243  
gr. (extraordinary dirty loaded)
  - N° of caps used: 1 (suggested dose); 2 ; 3.

15

In the Fuzzy Logic Washing machine the situation is more critical than in the Conventional washing machine, as the water in which the film and the boron can dissolve is less. The risk of finding residues at the end of the washing cycle increases with the following parameters:

increases with the following parameters:

- The decreasing of temperature ( from 60 to 30 °C)
  - The increasing of detergent dose
  - The increasing of number of caps used

Fuzzy	100 %	100 %	100 %	100 %	100 %	100 %	100 %	100 %	100 %
Logic	no resid								
	ue								

The red squares are those situations in which PVOH caps are likely (30-40% of cases) to give residues. With HPMC caps we  
5 never found residues even in the worse cases.

#### COMPATIBILITY WITH THE FORMULATION

The formulation in the HPMC film is perfectly stable: both  
10 the chemical-physical parameters and the stability and activity of the active ingredient in the formulation.

Stability tests at different conditions have been conducted on the HPMC caps.

Storage conditions (at all conditions gel caps were placed  
15 inside a PE (10/bag) inside a cartoon boxes- PE bags were stored closed and also open)

- 2 °C dry oven
- RT
- 30 °C/70% RH
- 20 ▪ 40 °C/75% RH
- 60 °C dry oven

Parameters checked:

- water up-date
- capsule resistance
- 25 ▪ product leakage/ diffusion

The capsule resistance is in line with the initial resistance

## CLAIMS

1. A water-soluble container comprising at least one compartment prepared from a thermoformed film of hydroxy propyl methyl cellulose (HPMC).  
5
2. A process for preparing a water-soluble container comprising at least one compartment which comprises:
  - a. feeding at least one HPMC film into a thermoforming machine;  
10
  - b. heating the at least one HPMC film to a temperature of 120 to 140 °C, ideally 125 to 135 °C, for 1 to 10 seconds, ideally 2 to 6 seconds;
  - c. simultaneously or subsequently forming the at least one  
15 heated HPMC film into a mould, which is preferably cooled (below room temperature, ideally below 20 °C);
  - d. filling the formed film pocket with at least one liquid or solid composition; and
  - e. sealing the formed and filled film pocket with a water-  
20 soluble film, preferably a HPMC film.

## ABSTRACT

## IMPROVEMENTS IN OR RELATING TO COMPOSITIONS

5      The present invention relates to a water-soluble container comprising at least one compartment prepared from a thermoformed sheet of hydroxy propyl methyl cellulose (HPMC) and to a process for preparing such a container.



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